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ANALYSIS OF DATA REDUCTION STRATEGY USED IN TA INSTRUMENTS Q800 DMA TEST SYSTEM

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Interim Report

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14. ABSTRACT
The Composites Branch of the Air Force Research Laboratory's Materials and Manufacturing Directorate (AFRL/RXCC) has observed anomalies in Q800 Dynamic Mechanical Analyzer (DMA) testing of fiber-reinforced polymer matrix composites related to both temperature dependence of the storage and loss moduli and to dependency of these data on specimen thickness using the dual-cantilever beam (DCB) clamp. Two factors are examined in detail: shear area factors used in the DCB stiffness calculations and coefficient of thermal expansion mismatch between the test specimen and the test frame. It is hoped this document will foster technical discussion with original equipment manufacturers of DMA instruments.

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dynamic mechanical analysis (DMA), fiber-reinforced polymer matrix composites, Q800 DMA, dual-cantilever clamp

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EXECUTIVE SUMMARY

It is the intent of this document to foster technical discussion with the original equipment manufacturer of the Q800 Dynamic Mechanical Analyzer (DMA) and materials engineers in the Composites Branch of the Air Force Research Laboratory's Materials and Manufacturing Directorate (AFRL/RXCC). The AFRL/RXCC has observed anomalies in DMA testing of fiber-reinforced PMCs related to both temperature dependence of the storage and loss moduli and to dependency of these data on specimen thickness using the dual-cantilever beam (DCB) clamp. Two factors are examined in detail: shear area factors used in the DCB stiffness calculations and CTE mismatch between the test specimen and the test frame.

1.0   INTRODUCTION

This is an informal document intended to serve as a basis for technical discussions between TA Instruments and engineers in the Composites Branch (RXCC) of the Materials and Manufacturing Directorate of the Air Force Research Laboratory (AFRL). The AFRL/RXCC routinely uses the Q800 DMA system to perform characterization of advanced fiber-reinforced composite materials for use in advanced models of thermo-chemical and thermo-mechanical behavior. In recent studies[1] we have observed anomalous responses in DMA tests using the double cantilever beam (DCB) specimen configuration. A parallel effort using the same approach is also addressing similar anomalies seen using the standard 50 mm three-point bend (3PB) clamp configuration but will not be reported here. The focus of the DCB apparent anomalies are related to both temperature dependence of the storage and loss moduli and to dependency of these data on specimen thickness. We have begun to perform systematic studies of the test procedure to assess the relative importance of several potentially significant factors. To date, we have examined only a subset of the possible factors that appear to affect the accuracy of the measured data or its interpretation and have noticed a few items that are worthy of some discussion. This note will focus on two issues that seem relatively uncomplicated, but which may affect the data we are collecting in a significant way.
  (1)   Shear area factors used in the DCB stiffness calculations are appropriate for isotropic materials, but not for composites. Assumed values are embedded in the test system software or firmware, and may cause reported modulus values to be significantly in error.
  (2)   Coefficient of thermal expansion (CTE) mismatch between the test specimen and the test frame may produce large artificial modulus variations in high-temperature tests.

2.0   EXPERIMENTAL

2.1   DCB Shear Factors

In the data reported by the Q800 system, beam stiffness values computed from the deflection amplitude and applied load are used first to define a stiffness (load/deflection), and then converted to storage modulus using a geometry factor that accounts for the response characteristics of the DCB configuration. If the specimen is very thin, it behaves approximately like an ideal Bernoulli-Euler beam, and the measured deflection is essentially due to pure bending. As the thickness of the specimen increases, transverse displacement caused by the shear forces may become significant, and the geometry factor attempts to include this effect. We believe that the assumptions built into the geometry factor calculation within the Q800 test system software or firmware may be inappropriate for fiber-reinforced polymer matrix composite (PMC) materials.

Appendix A contains a summary of our understanding of the development of the geometry factor and modulus equations used in the Q800 system, based on shear-deformable beam theory[2]. The calculation of the geometry factor as it is performed in the Q800 test system may be inappropriate for a composite specimen. The ratio E/G, which can be estimated with reasonable accuracy for metallic, or even homogeneous polymeric specimens, as $2(1+\nu)$, may be quite different for a PMC material. Also, the value $\nu = 0.44$ apparently used in this calculation in the Q800 system is reasonable for some polymers, but not for either metals or reinforced composites.

As an example, we consider an 8HS weave composite tested recently at RXCC. The storage moduli reported using the standard data reduction procedure, Equation (A.15)[2],

$$E = \frac{K_s}{F_c}\left[\frac{L^3}{24I} + 2\alpha(1+\nu)\frac{L}{2A}\right]$$

with the shear factor $\alpha=6/5$ and effective Poisson's ratio 0.44, for specimens of thickness 0.5, 1.5, 2.5, and 4.5 mm, at temperatures 20°C, 316°C, and 343°C, are shown in the solid curves in Figure 1 below. Note the artificially low values obtained from the thicker specimens, which exhibit a much larger proportion of transverse shear deformation than the thin specimens. A 50 to 70 percent reduction in storage modulus is calculated for the thickest specimens in this example (Figure 1).
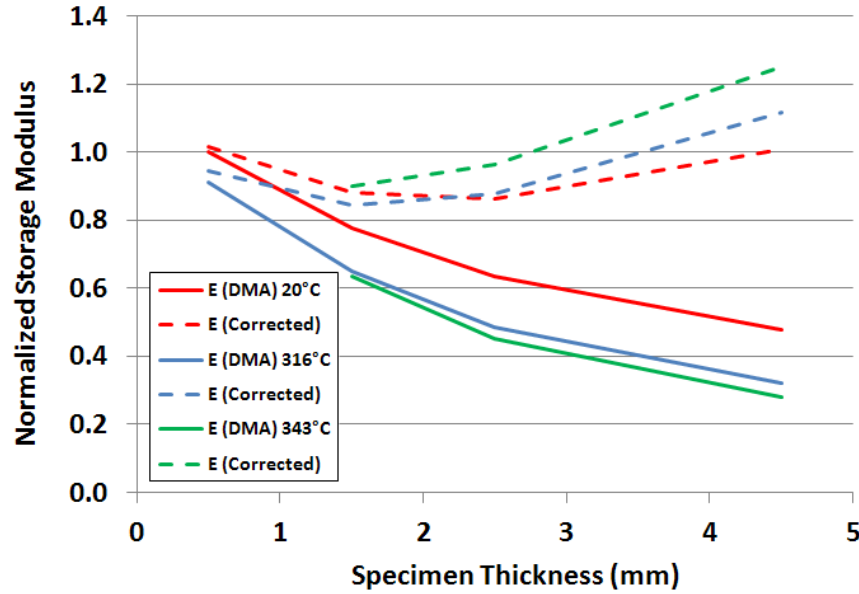
Figure 1. Reported moduli for 8HS weave, normalized with respect to room temperature value obtained from 0.5-mm specimen.

If we avoid the use of an effective Poisson's ratio in interpreting the measured data, the appropriate relationship for calculating the modulus is Equation (A.13)[2],

$$E = \frac{K_s}{F_c}\left[\frac{L^3}{24I} + \alpha\frac{E}{G}\frac{L}{2A}\right]$$

Obviously the use of this relationship presents a problem, because knowledge, or at least an estimate, of the ratio E/G is required. However, even a rough estimate of this parameter provides significantly better results for the composite modulus. Based on data for a similar 8HS weave[3], the ratio E/G varies from about 15 at room temperature to approximately 50 at 343°C. Using this additional information in the data reduction produces the dashed lines in Figure 1, which are significantly improved over the original data.

2.2 DCB Thermal Expansion Effects

The DMA test fixture is anchored to a metal base that is positioned in the bottom of the test chamber, and which is exposed to nominally the same temperature as the test specimen (Figure 2). The end and center supports of the DCB fixture are attached at their bases to the metal base, and above this point, the only connection between the supports is via the specimen, whose modulus and CTE are typically quite different from that of the base for a fiber-reinforced PMC. When the fixture and specimen are heated, differences in thermal expansion between the base and the specimen will result in potentially significant net tension in the specimen, and possibly additional bending deformation. A further possible side effect of this motion is a change in clamping forces that would affect the appropriate value of the clamping factor used in the stiffness calculation.

Figure 2. DCB test fixture including metal base.

AFRL/RXCC has performed preliminary finite element analyses (FEA) of the test configuration, including clamping forces and thermal expansion of the entire system, to assess the possible magnitude of these thermal effects. The specimen stiffness and modulus have been calculated from the finite element results using the same procedure outlined in the Q800 documentation (including the "standard" shear factor of $\alpha=6/5$). For two temperatures (23°C and 316°C) and four specimen thicknesses (0.5, 1.5, 2.5, 4.5 mm), Figure 3 shows the ratio of the modulus reported when thermal expansion is taken into account compared to that reported if thermal expansion of the base is eliminated. At room temperature, there is no effect; at elevated temperatures, the estimated error in the reported modulus can approach 20 percent. The modulus is underestimated for thinner specimens and overestimated for thicker ones.
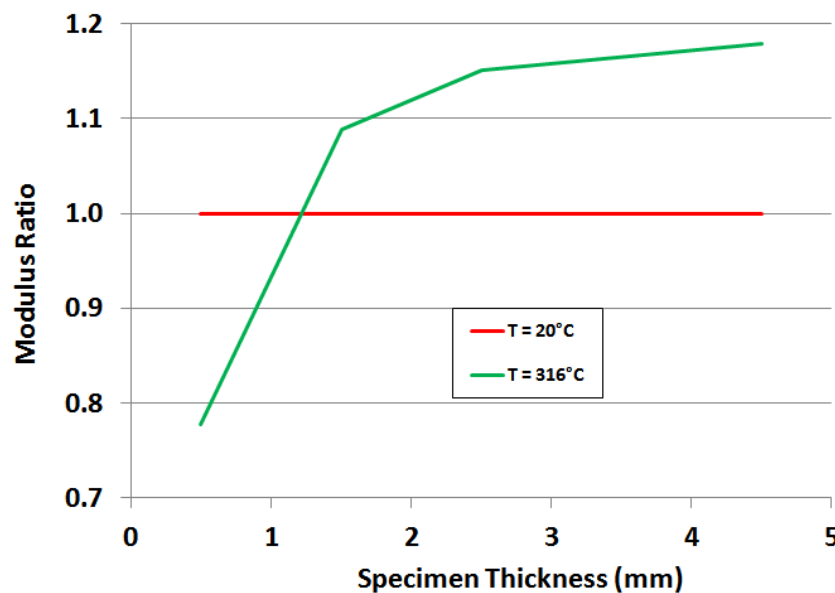
Figure 3. Approximate effect of fixture base thermal expansion on reported modulus.

3.0   CONCLUSIONS

We hope this paper fosters discussion and provides possible explanations for the anomalies seen in DMA testing of fiber-reinforced PMCs of various thicknesses using the DCB clamp. Specifically, it is believed that the effective Poisson's ratio of 0.44 and use of the elastic to shear modulus relationship used for the geometry factor calculation in the Q800 software contributes to the observed artifacts. In addition, the contributing effects of the bulky metal base thermal expansion on the reported modulus of the PMC specimens at elevated temperatures was analyzed via FEA modeling. While not quite as significant as the geometry factor calculation, the FEA results indicate that the modulus is notably underpredicted for thin specimens and overpredicted for thick specimens.

4.0 REFERENCES

1. Investigation of Glass Transition Temperature for Polyimide Composites: Study of Influence of Specimen Thickness and Thermo-Mechanical," *SAMPE Conference,* Long Beach, MD, 8 May 2013.

2. "Calculations Based on Clamp Type," *DMA Q Series HTML Help*, TA Instruments – Waters LLC, ©2001-2006.

3. K. S. Whitley and T. J. Collins, "Mechanical Properties of T650-35/AFR-PE-4 at Elevated Temperatures for Lightweight Aeroshell Designs," AIAA Paper No. 2006-2202, *47th AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics and Materials Conference*, Newport RI 2006.

APPENDIX A
Beam Theory Solutions

Classical Bending Solution for DCB

The deflection equation for a Bernoulli-Euler beam with no distributed load is $EI\dfrac{d^4v}{dx^4}=0$, for which the solution is a cubic polynomial $v(x)=a_0+a_1x+a_2x^2+a_3x^3$. For the dual cantilever beam, a typical half of the specimen has an imposed displacement $v(0)=\delta$ at its left end,
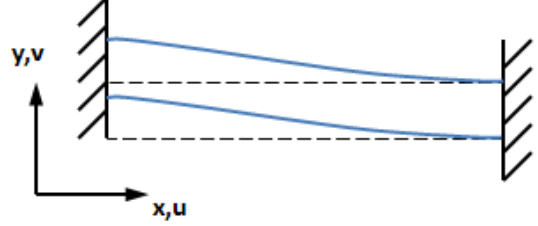
zero displacement at the opposite end, $v(L)=0$, and zero slope at both ends, $\dfrac{dv}{dx}(0)=\dfrac{dv}{dx}(L)=0$.

Applying these four boundary conditions gives the deflection solution for $v(x)$, which may be used directly to evaluate the remaining response variables of interest:

$$v(x)=\frac{\delta}{L^3}\left(2x^3-3Lx^2+L^3\right)$$

$$v'(x)=\frac{dw}{dx}=\frac{6\delta}{L^3}\left(x^2-Lx\right)$$

$$v''(x)=\frac{6\delta}{L^3}\left(2x-L\right)$$

$$v'''(x)=\frac{12\delta}{L^3}$$

$$M(x)=EIv''(x)=\frac{6EI\delta}{L^3}\left(2x-L\right)$$

$$V(x)=EIv'''(x)=\frac{12EI\delta}{L^3}$$

(A.1)

The applied force on this half of the specimen is simply $P=V(x)=12\,EI\delta/L^3$. The maximum moment, stress, and strain occur at each end, and are

$$M_{max}=\frac{6EI\delta}{L^2}=\frac{PL}{2}$$

$$\sigma_{max}=\frac{M_{max}t}{2I}=\frac{3Et\delta}{L^2}=\frac{3PL}{wt^2}$$

(A.2)

$$\varepsilon_{max}=\frac{\sigma_{max}}{E}=\frac{3t\delta}{L^2}$$

The bending flexibility, or displacement per unit applied force, is

$$\frac{\delta}{P}=\frac{L^3}{12EI}$$

(A.3)

Beam Stiffness and Flexibility including Shear Deformation

When the beam is relatively short compared with its depth, additional transverse displacements may occur that are associated with shear strains (short beam shear). The relevant shear strain is

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$$\gamma_{xy} = \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} \tag{A.4}$$

In classical (Bernoulli-Euler) beam theory, the rotation of the cross section, $\partial u / \partial y$, is equal and opposite to the slope $\partial v / \partial x$, so that the shear strain vanishes. For shear deformable beams, we account for the additional displacement by adding up the additional contribution to the deflection over the length of the beam. To obtain an estimate simply, we use the average shear strain at each cross section

$$\gamma_{xy}^{AVG} = \frac{V}{GA} \tag{A.5}$$

and scale it by a *shear area factor* that relates the variable shear strain within the cross section to the average. Normally the shear factor is chosen such that the shear strain energy, shear wave speed, or other macroscopic shear response characteristic is equal to the value obtained from a more detailed model of the beam. The resulting shear deflection contribution for the DCB is simply

$$\delta_{shear} = \int_0^L \alpha \frac{V}{GA} dx = \frac{PL}{GA} \alpha \tag{A.6}$$

Here α is the shear area factor. The combined deflection of the beam is the sum of the bending and shear displacement contributions,

$$\delta_{total} = \delta_{bending} + \delta_{shear} = \frac{PL^3}{12EI} + \frac{PL}{GA} \alpha = \frac{PL^3}{12EI}\left[1 + \alpha \frac{E}{G}\left(\frac{t}{L}\right)^2\right] \tag{A.7}$$

The corresponding beam stiffness that can be obtained from measurements in the DMA test is (considering only one beam, which is half the experimental specimen):

$$K_s = \frac{P}{\delta_{total}} = \frac{12EI}{L^3\left[1 + \alpha \frac{E}{G}\left(\frac{t}{L}\right)^2\right]} \tag{A.8}$$

In practice the measured value of K_s is modified to account for the effects of clamped boundary conditions that are not realized perfectly in the experiment, giving a corrected stiffness K defined by

$$K = K_s / F_c \tag{A.9}$$

This corrected stiffness replaces the measured value for purposes of further data reduction, so we have

$$K = \frac{K_s}{F_c} = \frac{12EI}{L^3\left[1 + \alpha \frac{E}{G}\left(\frac{t}{L}\right)^2\right]} \tag{A.10}$$

In the Q800 manuals, F_c is called the *clamping factor* and is obtained using a fit to finite element analysis results for various combinations of length and thickness,

$$F_c = 0.7616 - 0.02713\sqrt{L/t} + 0.1083\ln(L/t) \tag{A.11}$$

The details of the finite element calculations leading to this approximation of the clamping factor are not covered in the documentation, but presumably the analyses account for the differences between idealized beam bending boundary conditions and the actual contact constraints and shear lag that occur at the grips, and possibly the effects of rounded edges of the grips, and similar fine-scale details.

The calculation of the stiffness and modulus in the DMA system uses Equation (A.10) directly, with one adjustment; the value of K_s (or K) is defined based on the total force from both halves of the experiment, so the factor of 12 in the numerator of the right side is doubled. Therefore

$$K = \frac{K_s}{F_c} = \frac{12EI}{L^3\left[1+\alpha\dfrac{E}{G}\left(\dfrac{t}{L}\right)^2\right]} \tag{A.12}$$

The resulting modulus equation is

$$E = \frac{K_s}{F_c}\frac{L^3}{24I}\left[1+\alpha\frac{E}{G}\left(\frac{t}{L}\right)^2\right] = \frac{K_s}{F_c}\left[\frac{L^3}{24I}+\alpha\frac{E}{G}\frac{L}{2A}\right] \tag{A.13}$$

It is worth noting that, for an isotropic material obeying the relation $G = E/2(1+\nu)$, equations (A.12) and (A.13) become

$$K = \frac{24EI}{L^3\left[1+2\alpha(1+\nu)\left(\dfrac{t}{L}\right)^2\right]} \tag{A.14}$$

$$E = \frac{K_s}{F_c}\left[\frac{L^3}{24I}+2\alpha(1+\nu)\frac{L}{2A}\right] \tag{A.15}$$

which are the same forms appearing in the Q800 documentation (compare Equation A.14 with the expression for K on page B-1, and Equation A.15 with the expression for the geometry factor GF = E/K on page B-4).

The shear factor used in the stiffness expression (page B-1) appears to correspond to $\alpha = 6/5$, which is the value one obtains by equating the shear strain energy per unit area in the simplified model to the actual energy based on a parabolic shear stress distribution through the thickness, for a rectangular section and isotropic material. In the modulus equation (page B-4), the shear factor S corresponds to 2α, and the nominal value listed (S = 1.5) would imply $\alpha = ¾$. The second of these (S = 1.5) does appear to be used in computing the reported data values from the Q800 system.

The value of Poisson's ratio used in the GF calculation is listed (page B-4) as being 0.44, and this value does appear to be embedded in the GF used to compute the Q800's reported data.

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The material below is reproduced from the Q800 electronic documentation for reference[3].

Sample Stiffness and Modulus Calculations

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Dual Cantilever Equations

When running experiments using the dual cantilever clamp, the equations found in this section are applied to obtain your results.

Modulus Equation

The stiffness model equation for a rectangular cross section sample, analyzed on the dual cantilever clamp, is as follows. (Similar calculations were performed for cylindrical samples but are not detailed here.)

$$K = \frac{24 \cdot E \cdot I}{L^3 \cdot \left[1 + \frac{12}{5} \cdot (1 + v) \cdot \left(\frac{t}{L}\right)^2\right]}$$

Where:

K = stiffness or spring constant

E = elastic modulus

L = sample length (one side)

t = sample thickness

I = sample moment of inertia

v = Poison's ratio

The sample moment of inertia is

$$I = \frac{w \cdot t^3}{12}$$

Where:

t = sample thickness

I = sample moment of inertia

w = sample width

The stiffness model equation assumes that the ends of the sample are fixed, or that there is no deformation of the sample beyond where the sample enters the clamps. This is never achieved in practice, to do so would require a discontinuity in the strains within the sample at the clamp face. To account for this error, a sample stiffness correction factor can be defined as:

$$F_c = \frac{K_s}{K}$$

Where:

K = stiffness or spring constant

K_s = measured stiffness

F_c = clamping correction factor

Substituting for K in the model equation and solving for the modulus:

$$E = \frac{K_s}{F_c} \cdot \frac{L^3}{24 \cdot I} \left[1 + \frac{12}{5} \cdot (1 + v) \cdot \left(\frac{t}{L}\right)^2 \right]$$

$$Fc = 0.7616 - 0.02713 \; x \; \sqrt{\frac{L}{t}} + 0.1083 \ln \left(\frac{L}{t}\right)$$

Where:

E = elastic modulus

L = sample length (one side)

ln = natural log

K_s = measured stiffness

t = sample thickness

I = sample moment of inertia

v ☐☐= Poison's ratio

F_c = clamping correction factor

The clamping correction factor, F_c, was determined by finding the sample stiffness using *Finite Element Analysis* of the sample deformation and calculating F_c using the sample stiffness equation and the FEA stiffness. This was done by studying many cases including a variety of materials and geometries with corresponding fit applied to the result.

> NOTE: Poisson's ratio accounts for the shear deformation taking place in flexure, when using samples of relatively small length-to-thickness ratios. It is introduced in the equation using the standard equation relating E and G: E = 2 (1 + v) G

Stress and Strain

When using the dual cantilever clamp, stress and strain are not constant throughout the sample thickness. The maximum level of strain occurs at the sample surface, while the center experiences no strain at all. This also means that both stress and strain can have positive or negative sense, depending on whether it is on the top or bottom surface of the sample. The following equation expresses the maximum stress and strain levels and does not include any contribution from the clamp:

$$\sigma_x = \frac{3 \cdot P \cdot L}{w \cdot t^2}$$

$$\varepsilon_x = \frac{3 \cdot \delta \cdot t \cdot F_c}{L^2 \cdot \left[1 + \frac{12}{5} \cdot (1 + v) \cdot \left(\frac{t}{L}\right)^2 \right]}$$

Where:

σ_x = stress

ε_x = strain

P = 1/2 applied force

δ = amplitude of deformation

L = sample length (one side)

t = sample thickness

w = sample width

F_c = clamping correction factor

v = Poison's ratio

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Operating Range of Single/Dual Cantilever Clamps

The two equations below can help you determine which sample clamps to use and which sample sizes to choose. These equations can also help determine if the properties of a sample of a particular size can be measured or if the sample will have to be resized.

Shown below are the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equations below. The modulus range is based on the range of stiffness over which the DMA can operate (10^2 to 10^7 N/m).

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Geometry Factor Equations

Single Cantilever:

$$GF = \frac{1}{F}\left[\frac{L^3}{12I} + 2S(1+v)\frac{L}{A}\right]$$

Dual Cantilever:

$$GF = \frac{1}{F}\left[\frac{L^3}{24I} + S(1+v)\frac{L}{2A}\right]$$

where:

L = sample length (mm)

A = sample cross sectional area (mm^2)

I = geometric moment (mm^4) = 1/12 T^{3}W for rectangular samples

T = sample thickness (mm)

W = sample width (mm)

F = clamping factor (nominally 0.9)

S = shearing factor (nominally 1.5)

v = Poisson's ratio (nominally 0.44).

For more detail on the equations see Calculations Based on Clamp Type.

LIST OF ACRONYMS, ABBREVIATIONS, AND SYMBOLS

AFRL   Air Force Research Laboratory
CTE   coefficient of thermal expansion
DCB   dual-cantilever beam
DMA   dynamic mechanical analysis
DTIC   Defense Technical Information Center
FEA   finite element analysis
PMC   polymer matrix composite
RX   Materials and Manufacturing Directorate
RXCC   Composites Branch
3PB   three-point bend